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PCT

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**(54) Title:** POLYESTER FILM BASE FOR MOTION PICTURE FILM

**(57) Abstract**

A polyester resin is prepared by condensing dimethyl terephthalate and 1,4-cyclohexanedimethanol where the polyester has an inherent viscosity of less than 0.80 dl/gm in a 60:40 phenol/tetrachloroethane solvent blend and the 1,4-cyclohexanedimethanol reactant contains 75 to 100 % of its cis-isomer. This resin can be melt-cast to a film support suitable for motion picture film. Such film is particularly advantageous, because it can be formed without cracking, easily cut and perforated, will not be deleteriously affected by the presence of dust and splinters, and is made without adverse environmental effects.

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## POLYESTER FILM BASE FOR MOTION PICTURE FILM

FIELD OF THE INVENTION

This invention relates to a polyester resin prepared by condensing either terephthalic acid or dimethyl terephthalate and 1,4-cyclohexanedimethanol which is useful as a support for motion picture film.

5

BACKGROUND OF THE INVENTION

For many years, solvent-based cellulose triacetate film has been used as a photographic film support. Its physical characteristics and the dimensional uniformity and surface quality imparted by solvent casting have made cellulose triacetate the first choice for many photographic films.

Despite the excellent photographic properties of solvent-cast cellulose triacetate film, environmental concerns about solvents conventionally used in the casting of the films have created a need for a new method of manufacturing of cellulose triacetate films or for a new kind of photographic film support. Unfortunately, cellulose triacetate cannot be melt-cast, because its melting point is above its decomposition temperature. As for solvent casting of cellulose triacetate, no new solvents have been found which are more acceptable than the conventional ones.

One possible way to eliminate solvents is to melt cast a thermally stable polymer such as poly(ethylene terephthalate). Indeed, this type of polymer is used commercially for the manufacture of supports for photographic sheet films such as x-ray films and graphic arts films. It is not suitable, however, for many kinds of photographic films. As a motion picture film support, poly(ethylene terephthalate) is particularly unsuitable, because when poly(ethylene terephthalate) film is cut into strips and

punched with perforations, the film splinters and crumbles. As a result, such film has a high degree of particulate contamination. Moreover, poly(ethylene terephthalate) film is so tough that when punching and cutting it, punches and knives for such operations are rapidly worn. Consequently, manufacturing equipment must be shut down to replace the punches and knives. Still another problem with regard to the use of a poly(ethylene terephthalate) film base for motion picture films is that the toughness of the film prevents the film from breaking under certain conditions where film breakage is preferable to the damage the projector will otherwise suffer. Therefore, poly(ethylene terephthalate) film is difficult to use in motion picture films.

Esters of cellulose hydroxyl groups made over a wide range with both single and mixed acids have been used for a variety of different purposes. Cellulose diacetate ( $DS_{ac} = 2.45$ ), unlike the triacetate, has a sufficiently low melting point that, with adequate plasticizer addition, it can be melt extruded. Mixed esters, or triacetates having their acetyl groups replaced with propionic or butyric groups can accomplish the same purposes. However, such cellulose ester compositions with lower acetyl content than the triacetate are deficient in properties that are necessary for photographic roll film supports, most notably stiffness and heat distortion temperature. In addition, the plasticizers and flame retardants used in commercial melt-processable cellulose esters are volatile at extrusion temperatures and generate aerosols which can condense and create surface defects on the film.

U.S. Patent No. 2,901,466 to Kibler ("Kibler") discloses linear polyesters prepared by condensing 1,4-cyclohexanedimethanol with one or more bi-functional

reactants, including terephthalic acid. Kibler suggests using such polymers as photographic film supports in general but makes no mention of motion picture film in particular. Both cis- or trans-1,4-cyclohexanedimethanol are disclosed by Kibler but, when large quantities (over 50%) of the cis-isomer are utilized, the resulting polymer has a very high inherent viscosity. As a result, films made from these polyesters have a higher percentage of break elongation which makes them more difficult to cut or perforate. Kibler's polyester films have lower inherent viscosities only when a low cis-isomer level is employed. Films with such cis-isomer levels are unsuitable for motion picture film, because they have high crystallization temperatures, making them difficult to orient biaxially without cracking.

In view of the above-discussed deficiencies of currently-available film, there remains a great need for a melt-castable film having properties suitable for photographic film supports, particularly for motion picture film.

#### SUMMARY OF THE INVENTION

The present invention relates to a polyester film prepared by condensing dimethyl terephthalate and 1,4-cyclohexanedimethanol where the resulting polyester has an inherent viscosity of preferably less than 0.80 decaliter ("dl")/gram in a 60:40 phenol/tetrachloroethane solvent blend and the 1,4-cyclohexanedimethanol contains 75% to 100% of its cis-isomer. This resin is useful for forming a polyester film support for photographic films, particularly motion picture film.

The polyester films of the present invention are readily slit, without dulling knives, to widths suitable for photographic film. Further, the edges of

the film are perforated without dulling the operative portion of the punching device. Due to the ease with which such films are cut and perforated, the resulting motion picture film is generally free from impurities caused by cracking and splintering. In addition to these advantages, such polyester films can be cast without creating environmental hazards.

#### DETAILED DESCRIPTION OF THE INVENTION

10 In preparing the polyester resins of the present invention, dimethyl terephthalate or terephthalic acid and 1,4-cyclohexanedimethanol are polymerized by conventional condensation reactions which are well known in the art. Typically, this reaction  
15 involves reacting the dimethyl terephthalate and 1,4-cyclohexanedimethanol in a vessel by heating the vessel contents to a temperature of 200° to 220°C, preferably 220°C, to melt the reactants. After melting is completed, the vessel's contents are further heated  
20 to 285° to 295°C, preferably 290°C. Polycondensation is carried out at 290°C using a pressure of less than 0.3 mm Hg for 25 to 35 minutes. The reaction is then terminated.

Dimethyl terephthalate or its acid derivative  
25 is conventionally available from a variety of sources. 1,4-cyclohexanedimethanol is also commercially available from Eastman Chemical Co., Kingsport, Tennessee under the trade name EASTMAN CHDM-D. However, such commercial grades of 1,4-cyclohexanedimethanol contain about 30% of  
30 the cis-isomer and about 70% of the trans-isomer. The cis-isomer is recovered by dissolving this mixture in a solvent to crystallize out the trans-isomer. The remaining cis-1,4-cyclohexanedimethanol is separated by vacuum distillation to yield 90 to 95%, preferably 95%,  
35 cis-1,4-cyclohexanedimethanol. The crystallization solvent can be ethyl acetate and other similar solvents.

such as isopropanol and diethyl ether. The resulting 1,4-cyclohexanedimethanol has 75 to 100%, preferably 90 to 95%, of the cis- isomer with the balance being the trans- isomer.

5       The condensation reaction is advantageously facilitated by use of a catalyst. Suitable catalysts include titanium tetraisopropoxide, acetyl triisopropyl titanate, dibutyltin diacetate, butyltin stannic acid, dibutyltin dilaurate, gallium lactate, germanium dioxide  
10 calcium acetate, and manganese diacetate with titanium tetraisopropoxide being particularly preferred. Such catalysts are usually dispersed in a solvent with the solvent composition containing 0.20 to 1.50, preferably  
0.50, percent of the dispersed catalyst. Suitable  
15 solvents include butanol, ethylene glycol, acetone and mixtures thereof. When catalytically promoting the condensation reaction of the present invention, the solvent with dispersed catalyst is added to the reaction vessel at the same time as the reactants.

20       The molar ratio of the dimethyl terephthalate and 1,4-cyclohexanedimethanol reactants is 1:1.20 to 1:1.50, preferably 1:1.25. When using terephthalic acid, the ratio should be 1:1.10 to 1:1.40, preferably 1:1.20. Although dimethyl terephthalate and  
25 1,4-cyclohexanedimethanol theoretically react in a 1:1 molar ratio, it is preferable to utilize an excess of 1,4-cyclohexanedimethanol to facilitate complete ester exchange or to obtain high conversion. When such reaction ratios are utilized under the above reaction  
30 conditions, the number average molecular weight of the resulting polyester is 20,000 to 35,000 grams/mole, preferably 25,000 grams/mole.

The resulting polyester has an inherent viscosity of less than 0.80, preferably less than 0.65,  
35 dl/gm in a 60:40 phenol/tetrachloroethane solvent



blend. In addition, the polyester should have a crystalline melt point of 230°C to 295°C, preferably 240°C.

The resulting polymer from the above-discussed  
5 condensation reaction, is ground, pulverized, or  
pelletized to a form from which a film of such material  
can be produced. Techniques of forming films from such  
polyester material are well known in the art. In such  
processes, the polyester is first charged to a screw  
10 conveyor where they are heated to a melt at a  
temperature of 230°C to 315°C, preferably 270°C. The  
screw then conveys the melt through an extruder die from  
which it drops on to a revolving polished drum as a  
film. The drum is cooled so that as the film passes  
15 around the drum, the film is quenched to produce a clear  
sheet. Generally, the extruded film has a melt  
temperature of 230°C to 320°C, preferably 270°C, when it  
first contacts the cooled drum, and the cooled film  
separates from the drum at a quenching temperature of  
20 20°C to 100°C, preferably 80°C. A significant advantage  
of the polyester resin of the present invention is that,  
when it is cast, the resulting film has a low  
crystallization temperature (i.e., less than 180°C,  
preferably 158°C) compared to such polyesters with lower  
25 cis- isomer contents. The lower crystallization  
temperature of the polyester films of the present  
invention causes them to begin crystallizing at a later  
point in their travel around the cooling drum. With  
drum speed, quenching temperature, and film-drum  
30 separation locations all being constant, the polyester  
film of the present invention undergoes crystallization  
for less time (and, therefore, to a lesser extent) than  
films made from resins with lower levels of the cis-1,4-  
cyclohexan dimethanol isomer. As a result, the  
35 polyester films of the present invention can be  
substantially biaxially oriented without becoming too  
brittle.

After the film leaves the cooling drum, it must be biaxially stretched to a thickness of 2 mils to 7 mils, preferably 5.6 mils. Biaxially stretching requires that the film be simultaneously stretched in both the transverse and machine directions using well-known techniques or separately and sequentially treated in this fashion. When separately stretching a film in these directions, it can be stretched in the transverse and machine directions in either order. In the transverse direction, the film is stretched by 3 to 5 times, preferably 3, times, while it is stretched in the machine direction by 2 to 4 times, preferably 3.4, times. During such stretching, the film is maintained at a temperature of 80°C to 140°C, preferably 80 to 120°C.

Following biaxial stretching, the film's stretched dimensions are set by heating it to a temperature of 175°C to 240°C, preferably 200 to 220°C, for 2 to 20 seconds, preferably 4 seconds.

The resulting film has properties which enable it to be cut and punched without undue force. These properties include: a percentage of elongation of 20 to 50%, preferably less than 40% and a cut force of less than 50 lbs., preferably less than 40 lbs.

In the form of a film, the polyester resin of the present invention is suitable as a support for photographic film, particularly motion picture film. Such photographic film is prepared by melting a dispersion of a photographically-sensitized gelatin, applying the gelatin to the surface of the film, and allowing the sensitized-gelatin to solidify. Generally, a plurality of layers should be applied. The photographic film can then be cut into strips of suitable width and perforated without dulling knife blades or punching devices.

EXAMPLES

Example 1 - Polyester of 95%/5% cis/trans 1,4-cyclohexanedimethanol and dimethyl terephthalate.

2,000 grams of a 30%/70% cis/trans mixture of 1,4-cyclohexanedimethanol, commercially available under the trademark EASTMAN CHDM-D was dissolved in ethyl acetate. After the trans- isomer was crystallized out of solution, the remaining ethyl acetate-cis/trans mixture of 1,4-cyclohexanedimethanol was separated by vacuum distillation leaving a product comprising 95% cis-1,4-cyclohexanedimethanol and 5% trans-1,4-cyclohexanedimethanol.

To a 500 ml single-neck flask was charged 97.0g (0.5 mole) of dimethyl terephthalate, 90.0g (0.5 mole plus 25 mole percent excess) of the 1,4-cyclohexanedimethanol (95% cis), and 0.99 ml of a 1.04% solution of titanium tetraisopropoxide in n-butanol. The flask, fitted with a metal stirrer, a nitrogen inlet, and a volatiles outlet, was immersed in a Belmont metal bath at 220°C. The mixture was heated and stirred under a nitrogen blanket at 220°C until a clear solution was obtained. The bath temperature was then increased from 220° to 290°C over a period of 20 minutes. The reaction was then subjected to a vacuum (<0.3 mmHg) for 30 to 40 minutes and returned to atmospheric pressure with nitrogen. After cooling to ambient temperature, the resulting white opaque polymeric solid was then removed from the flask.

The inherent viscosity of the polymer was measured by adding 0.5 grams of it to a 125 ml Erlenmeyer flask and then adding 100 ml of a 60%/40% mixture of phenol/tetrachloroethane. The flask was placed on a laboratory hot plate and heated from 23°C to 125°C while being stirred for a period of 10 minutes. The flask was placed on a second hot plate and held at 125°C for 5 minutes. The mixture was cooled to 23°C and

filter d. The solution was passed through a modified Wagner capillary viscometer at 23°C, which showed an inherent viscosity of .78 dl/gm in a 60:40 phenol/tetrachloroethane solvent blend.

- 5           The polyester was also analyzed to determine its crystalline melting point. This analysis involved adding 10 to 20 mg of the polymer (ground to pass through a 4 mm screen) to an aluminum pan, sealing the pan, placing it in the sample head of a DuPont
- 10 Instruments 912 Differential Scanning Calorimeter ("DSC") fitted with a DSC autosampler. The first DSC cycle scan was started at 23°C and heated at 20°/min. to 350°C under a nitrogen sweep of 120 cc/min. After the sample was quenched and cooled to room temperature,
- 15 second and third DSC cycle scans were conducted. DSC traces were then plotted using a DuPont 9900 Computer - Thermal Analyzer. As a result of this processing, the polyester was found to have a crystalline melting point of 251°C, a crystallization temperature on heating of
- 20 120°C, a crystallization temperature on cooling of 168°C, and a glass transition point of 76.0°C.

Poly(ethylene terephthalate) film sold under the trade name ESTAR sold by Eastman Chemical Co., Kingsport, Tennessee has a crystallization temperature

25 on cooling of 167°C. Consequently, the resin made by the process of Example 1 achieves the same low level of crystallization (and, consequently, the same low level of film cracking) as the poly(ethylene terephthalate) resin widely used in the film-making industry. As

30 demonstrated below, the resins of Examples 2-4, which contain higher levels of trans-1,4-cyclohexanedimethanol, do not achieve such favorable results, because they have higher degree of crystallinity, a higher melting point, and a higher

35 crystallization temperature on cooling.

Example 2 - Polyester of 60.1%/39.9% cis/trans  
1,4-cyclohexanedimethanol and dimethyl terephthalate  
60.1%/39.9% cis/trans 1,4-cyclohexanedimethanol  
was obtained by mixing 300 grams of the  
5 commercially-available 30%/70% cis/trans  
1,4-cyclohexanedimethanol used as a starting material in  
Example 1 with 300 grams of the 95%/5% cis/trans  
1,4-cyclohexanedimethanol product of Example 1.

The polymerization process described in  
10 Example 1 was then repeated except that 90.0 grams of  
the 60.1%/39.9% cis/trans 1,4-cyclohexanedimethanol was  
used instead of the 95%/5% cis/trans  
1,4-cyclohexanedimethanol and the polycondensation  
reaction temperature was raised from 220° to 305°C  
15 instead of from 220°C to 290°C.

The resulting white opaque solid polyester had  
a crystalline melting point of 260°C, a crystallization  
temperature on cooling of 186°C, and an inherent  
viscosity of 0.74 dl/gm in a 60:40  
20 phenol/tetrachloroethane solvent blend.

Example 3 - Polyester of 60.3%/39.7% cis/trans  
1,4-cyclohexanedimethanol and dimethyl terephthalate  
60.3%/39.7% cis/trans 1,4-cyclohexanedimethanol

25 was prepared in substantially the same fashion as the  
60.1%/39.9% cis/trans blend of Example 2. The  
polycondensation process described in Example 1 was then  
repeated. The polycondensation reaction temperature was  
kept at 305°C. The resulting white opaque solid  
30 polyester had a crystalline melting point of 257°C, a  
crystallization temperature on cooling of 169°C, and an  
inherent viscosity of .75 dl/gm in 60:40 a  
phenol/tetrachloroethane solvent blend.

35 Example 4 - Polyester of 29.1%/70.9% cis/trans  
1,4-cyclohexanedimethanol and dimethyl terephthalate

Commercially available 30% cis/70% trans 1,4-cyclohexanedimethanol and dimethyl terephthalate were polycondensed at 310°C. The resulting white opaque solid polyester had a crystalline melting point of 294°C, a crystallization temperature on cooling of 240°C, and an inherent viscosity of 0.76 dl/gm in a 60:40 phenol/tetrachloroethane solvent blend.

From Examples 1-4, it is easily seen that the crystallization temperature on cooling decreases significantly with increases in cis-1,4-cyclohexanedimethanol content.

#### Example 5 - Film Manufacture

The polyester resins of Examples 1-4 were made into films by melting the resins to the temperatures in Table 1, extruding the melted resin in sheet form with a Brabender extruder on to a cool roller having a temperature according to Table 1, biaxially orienting the extruded sheet in a T.M. Long machine at the orientation temperatures and stretch ratios in Table 1, and heat setting the film for 2 minutes in a forced-air oven with a metal heatset frame at the temperatures shown Table 1.

TABLE 1

Resin	Extrusion Temp (°C)	Roller Temp (°C)	Orientation Temp (°C)	Stretch Ratio	Heat Set Temp (°C)
Ex.1 Polyester	276	75	95	3X by 3X	220
Ex.2 Polyester	315	105	104	3X by 3X	230
Ex.3 Polyester	285	105	104	3X by 3X	230
Ex.4 Polyester	315	105	110	3X by 3X	240

After these films were prepared, their properties were evaluated with an Instron tensile tester, and these results are set forth in Table 2.

TABLE 2

Film	Break Elongation	Young's Modulus of Elasticity (psi)	Toughness (in.-lb./ cubic inch)
5 Ex. 1 Polyester	27%	$5.3 \times 10^5$	8,100
Ex. 2 Polyester	44%	$4.4 \times 10^5$	13,900
Ex. 3 Polyester	34%	$4.7 \times 10^5$	10,300
Ex. 4 Polyester	39%	$4.8 \times 10^5$	12,600

10 The relatively low break elongation of the film made from the resin of Example 1 shows that it is less brittle than the other films and, therefore, less likely to generate dirt during cutting. The high Young's

15 Modulus of Elasticity of the film made from the polyester resin of Example 1 shows it is stiffer, thereby permitting the film to be made thinner and still achieve a desired stiffness. Finally, the relatively low toughness of the resin of Example 1 shows that it is

20 easier to cut.

These film samples as well as those produced from the poly(ethylene terephthalate) resin and either designated as C89-5 or sold by Eastman Chemical Co., Kingsport, Tennessee under the ESTAR trade name were

25 tested in a laboratory chopper. Samples of these films were placed on a lower stationary knife with the sample's forward end extending slightly beyond this knife edge. An upper knife, positioned relative to the lower knife to form a shearing zone, is driven

30 pneumatically at 15 inches per second toward the lower knife. A strain gauge attached to the lower knife monitors force readings. The distance travelled by the lower knife before the film cracks (i.e. knife elongation) is measured by an optical device. From the

35 cutting force and knife elongation, an energy level is calculated. Table 3 summarizes these results.

TABLE 3

Film	Cutting	Knife	En rgy
	Force (lbs.)	Elongation (mils)	(in.-lbs.)
5			
Ex. 1 Polyester	39.7	3.0	0.04
Ex. 2 Polyester	35.6	2.6	0.03
Ex. 3 Polyester	36.2	2.9	0.04
Ex. 4 Polyester	36.4	2.9	0.04
10 ESTAR	68.6	4.8	0.09
C89-5	65.1	6.2	0.12

The results of Table 3 show that films made from the resin of Example 1 (as well as those made from the resins of Examples 2-4) are much easier to cut than films made from poly(ethylene terephthalate).

Although the invention has been described in detail for the purpose of illustration, it is understood that such detail is solely for that purpose, and variations can be made therein by those skilled in the art without departing from the spirit and scope of the invention which is defined by the following claims.



## WE CLAIM:

- 1           1. A polyester resin prepared by condensing  
reactants consisting essentially of either terephthalic  
acid or dimethyl terephthalate and  
1,4-cyclohexanedimethanol, wherein said polyester has an  
5 inherent viscosity of less than 0.80 dl/gram in a 60:40  
phenol/tetrachloroethane solvent blend and said  
1,4-cyclohexanedimethanol is 75 to 100%  
cis-1,4-cyclohexanedimethanol.
- 1           2. A polyester resin according to claim 1,  
wherein the inherent viscosity is less than 0.65 dl/gram  
in a 60:40 phenol/tetrachloroethane solvent blend.
- 1           3. A polyester resin according to claim 1,  
wherein said 1,4-cyclohexanedimethanol is 94 to 100%  
cis-1,4-cyclohexanedimethanol.
- 1           4. A polyester film sheet formed from a  
polyester resin prepared by condensing reactants  
consisting essentially of either terephthalic acid or  
dimethyl terephthalate and 1,4-cyclohexanedimethanol,  
5 wherein said polyester has an inherent viscosity of less  
than 0.80 dl/gram in a 60:40 phenol/tetrachloroethane  
solvent blend and said 1,4-cyclohexanedimethanol is 75  
to 100% cis-1,4-cyclohexanedimethanol.
- 1           5. A polyester film according to claim 4,  
wherein the inherent viscosity is less than 0.65 dl/gram  
in a 60:40 phenol/tetrachloroethane solvent blend.
- 1           6. A polyester film according to claim 4,  
wherein said 1,4-cyclohexanedimethanol is 94 to 100%  
cis-1,4-cyclohexanedimethanol.

1           7. A polyester film according to claim 4,  
wherein said film has a crystallization temperature of  
less than 180°C.

1           8. A polyester film according to claim 4,  
wherein said film has a cut force of less than 40 lbs.

1           9. A polyester film according to claim 4,  
wherein said film has a toughness below 8500  
in.-lb./cubic inch.

1           10. A polyester film according to claim 4,  
wherein said film is biaxially oriented and heat set.

1           11. A polyester film according to claim 10,  
wherein said film has been stretched by 2.5 to 5.0 times  
in the transverse direction.

1           12. A polyester film according to claim 10,  
wherein said film has been stretched by 2.5 to 5.0 times  
in the machine direction.

1           13. A perforated motion picture film  
comprising:

5           a polyester film support prepared by  
condensing reactants consisting essentially of either  
terephthalic acid or dimethyl terephthalate and  
1,4-cyclohexanedimethanol, said polyester has an  
inherent viscosity of less than 0.80 dl/gm in a 60:40  
phenol/tetrachloroethane solvent blend and said  
1,4-cyclohexanedimethanol is 75 to 100%  
cis-1,4-cyclohexanedimethanol and

10           a coating over said support, wherein said  
coating comprises one or more silver halide-containing  
layers.

1           14. A motion picture film according to  
claim 13, wherein the inherent viscosity is less than  
0.65 dl/gm. in a 60:40 phenol/tetrachloroethane solvent  
blend.

1           15. A motion picture film according to  
claim 13, wherein said 1,4-cyclohexanedimethanol is 94  
to 100% cis-1,4-cyclohexanedimethanol.

1           16. A motion picture film according to  
claim 13, wherein said polyester film has a  
crystallization temperature of less than 180°C.

1           17. A motion picture film according to  
claim 13, wherein said polyester film has a cut force of  
less than 40 lbs.

1           18. A motion picture film according to  
claim 13, wherein said polyester film has a toughness  
below 8500 in.-lb./cubic inch.

1           19. A motion picture film according to  
claim 13, wherein said polyester film is biaxially  
oriented and heat set.

1           20. A motion picture film according to  
claim 13, wherein layers are layers of gelatin in which  
silver halide grains are dispersed.

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 92/01178

## I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all)<sup>6</sup>

According to International Patent Classification (IPC) or to both National Classification and IPC  
 Int.Cl.5 C 08 G 63/199 G 03 C 1/795

## II. FIELDS SEARCHED

Minimum Documentation Searched<sup>7</sup>

Classification System	Classification Symbols
Int.Cl.5	C 08 G G 03 C

Documentation Searched other than Minimum Documentation  
to the Extent that such Documents are Included in the Fields Searched<sup>8</sup>

## III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>9</sup>

Category <sup>10</sup>	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
X	US,A,2901466 (C.J. KIBLER et al.) 25 August 1959, see claims 1,6-9,26-28; column 1, lines 15-41; column 7, lines 27-32; example 8 (cited in the application)	1-7
Y		1-7
Y		10,13- 16,19, 20
X	--- J. Polym. Sci., Pol. Chem. Ed., vol. 11, no. 11, November 1973, R.W. LENZ et al.: "Crystallization-induced reactions of copolymers. III. Ester interchange reorganization of poly(cis/trans-1,4-cyclohexylenedimethylene terephthalate)", pages 2927-2946, see abstract; page 2930, figure 1; pages 2941-2945 --- -/-	1-3

\* Special categories of cited documents : <sup>10</sup>

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## IV. CERTIFICATION

Date of the Actual Completion of the International Search

16-06-1992

Date of Mailing of this International Search Report

9. 07. 92

International Searching Authority

EUR PEAN PATENT OFFICE

Signature of Authorized Officer

Maria Peis

Mark Pat

## III. DOCUMENTS CONSIDERED TO BE RELEVANT

(CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	Angew. Chem., vol. 74, no. 16, 7 July 1962, Dr. E.V. MARTIN et al.: "Struktur und Eigenschaften einer neuen Polyesterfaser [*]", pages 624-628, see page 626, figure 6 ---	7
Y	G.B. BUTLER et al.: "Reviews in Macromolecular Chemistry", 1973, vol. 9, J.R. MARTIN et al.: "Mechanical properties of polymers: the influence of molecular weight and molecular weight distribution", pages 57-199, see page 71, figure 8; page 77, figure 14; page 84, figure 18; pages 102-103, 105-109; page 106, figure 36 ---	1-7
Y	H.F. MARK et al.: "Encyclopedia of Polymer Science and Engineering", 1988, vol. 11, E. ARMOUR et al.: "Photographic applications", John Wiley & Sons, New York, US, pages 175-186, see pages 176-179 -----	10, 13-16, 19, 20

**ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO.**

**US 9201178**

**SA 57784**

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on 03/07/92. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 2901466		None	

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82